ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** 6D reconstruction of atomic strain in bulk metallic glasses	**Experiment number:** HC-2639
Beamline: ID15A	**Date of experiment:** from: 07.12.2016 to: 12.12.2016	**Date of report:** 31.08.2017
Shifts: 14	**Local contact(s):** Vaughan Gavin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

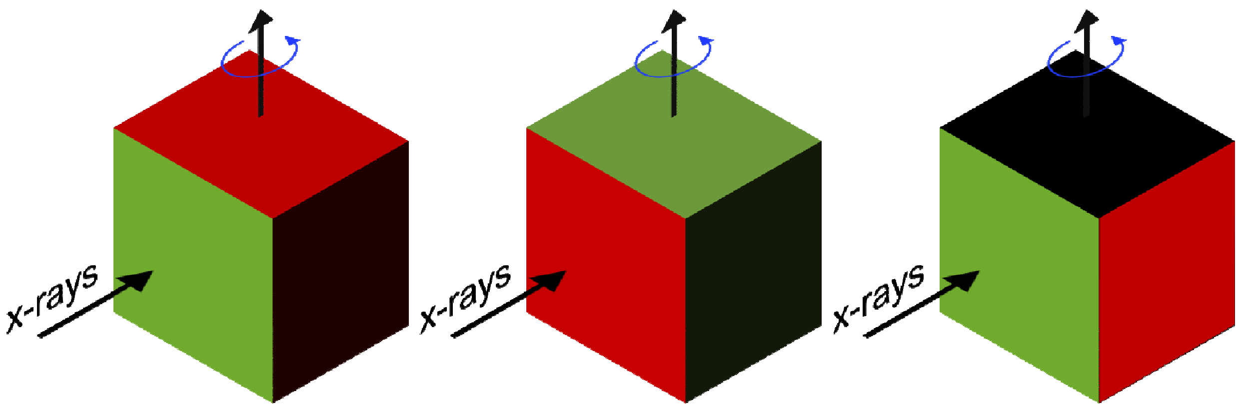
The lack of a periodic arrangement of atoms in bulk metallic glasses (BMGs) renders the detailed understanding of their elasto-plastic deformation mechanisms a very challenging task because structural studies cannot rely on a regular crystal structure and on the dynamics of related defects, such as dislocations. Strain analysis using high-energy XRD has been recently extensively used to overcome this limitation. Unfortunately, in this type of study planar averaged strains are assumed, with only a 2D strain tensor characterized. The overall picture is thus incomplete, as it assumes zero strain along the X-ray beam direction. The situation is further complicated by the fact that plastic deformation in BMGs is heterogeneous, occurring in highly localized shear bands, which precludes the possibility to simply rotate the samples by 90° to analyze the strain along the missing direction. To characterize the strain arising from different regions within the sample thus requires a tomographic approach, but standard tomographic reconstruction relies on the assumption of rotational invariance.

The aim of our experiments was to extend the strain analysis to 6D by tensor-resolved XRD computed tomography (XRDCT). To achieve this purpose, we used a recent approach able to spatially resolve preferred orientation via SAXS tomography using several tilt angles of the real tomographic axis in order to collect a set of projections, which may be regrouped to create a series of equivalent tomographies around a set of virtual axes [1]. As the SAXS data are slowly varying, the same projection may be sufficiently close to the ideal for use in several different virtual tomographic reference frames, meaning that a full data set may be collected in reasonable times. The problem we wish to address is computationally equivalent, the strain anisotropy manifesting itself in an azimuthal variation of the scattering maxima positions (or peaks in the real space G(r)), rather than intensity in the case of the SAXS data.

We tested the applicability of this technique to metallic glasses by first examining imprinted $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ BMGs. This material was selected because it displays macroscopically-strained areas of about 200-300 μm, suitable for relatively fast test tomography measurements by using a beam size of 15×25 μm^2. In addition, 2D strain maps of this particular sample have been already acquired [2], which allows us to compare them to the 3D strain maps from XRD tomography. We then performed a more challenging experiment aimed to investigate the strain in the elastic matrix around well-spaced shear bands generated by cold rolling of $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ BMG plates. This morphology consists of parallel shear bands forming an angle of about 45° with the rolling direction. The distance between the shear bands is ~50 μm.

Our experiment was organized as follows: initially, tomograms were acquired in a standard fashion, with a vertical tomographic axis with linear steps corresponding to the beam size, and a roughly equivalent number of angular projections over 180° at each position. For full 3D strain-tensor resolved measurements, an equivalent data collection was carried out at a number of tilt angles.

An additional set of experiments was performed in order to facilitate the reconstruction through the acquisition of redundant strain data. This approach, schematically shown in the figure below, was used to analyze both the imprinted and cold-rolled $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ BMGs. For the same sample, standard tomograms were acquired at different axes of rotation. For each axis of rotation, the reconstruction by standard methods leads to 3D maps of the out-of-plane and averaged in-plane strains. The redundancy of the data (two reconstructions will have one strain component in common) can then be used to obtain the complete 6D characterization.



All in all, we consider these measurements to have been a large success, as we have accomplished our goals of data acquisition outlined in the proposal. No experimental difficulties were encountered and the scientific support from the local contact was outstanding.

The complete data sets (20×20 or 100×100 projections per layer and per tilt) necessary for the complete 6D reconstruction required the acquisition of approximately 120k or 1M total patterns. Processing of such an amount of data is extremely time consuming. The work is currently in progress and we expect to complete the 6D strain reconstructions of the different samples within the end of the year.

[1] F. Schaff *et al.*, *Nature* **527** 353 (2015).
[2] S. Scudino *et al.*, *Appl. Phys. Lett.* **106**, 031903 (2015).